

LASER PHOTOLYTIC DEPOSITION OF THIN FILMS†

P. K. BOYER, C. A. MOORE, R. SOLANKI,* W. K. RITCHIE,* G. A. ROCHE** AND
 GEORGE J. COLLINS
 Department of Electrical Engineering, Colorado State University, Fort Collins,
 CO 80523

ABSTRACT

An excimer laser is used to photochemically deposit thin films of silicon dioxide, silicon nitride, aluminum oxide, and zinc oxide at low temperatures (100–350°C). Deposition rates in excess of 3000 Å/min and conformal coverage over vertical walled steps were demonstrated. The films exhibit low defect density and high breakdown voltage and have been characterized using IR spectrophotometry, AES, and C-V analysis. Device compatibility has been studied by using photodeposited films as interlayer dielectrics, diffusion masks, and passivation layers in production CMOS devices.

Additionally, we have deposited metallic films of Al, Mo, W, and Cr over large ($>5 \text{ cm}^2$) areas using UV photo-dissociation of trimethylaluminum and the refractory metal hexacarbonyls. Both shiny metallic films as well as black particulate films were obtained depending on the deposition geometry. The black films are shown to grow in columnar grains. The depositions were made at room temperature over pyrex and quartz plates as well as silicon wafers. We have examined the resistivity, adhesion, stress and step coverage of these films. The films exhibited resistivities at most ~ 20 times that of the bulk materials and tensile stress no higher than $7 \times 10^9 \text{ dynes/cm}^2$.

INTRODUCTION

There exists a need for low-temperature semiconductor fabrication processes to minimize wafer warpage, dopant redistribution, and defect generation and propagation [1]. Moreover, film deposition over photoresist for applications such as tri-level resists for high-resolution lithography and direct patterning via lift-off is desired at temperatures below $\sim 200^\circ\text{C}$ (above which resist degradation occurs) [2]. In this work we discuss a new low temperature, high deposition rate (up to 5000 Å/min) film growth technique which uses a excimer laser to photolyze gas-phase reactants whose products condense and form the desired film. This technique has been used to deposit dielectric films of SiO_2 , Si_3N_4 , Al_2O_3 and ZnO , and conducting films of Al, Cr, Mo, and W. The properties of these films are reviewed and compared to conventional deposition techniques.

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*NCR Microelectronics, Fort Collins, CO 80526

*Present Address: Johns Hopkins University, Department of Physics, Baltimore, MD 21218.

**Present address: Thermco Inc., Orange, CA 92668.

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EXPERIMENTAL APPARATUS

A Lumonics 860T excimer laser provides ultraviolet photons in a beam of rectangular cross-section which is down-collimated to a cross-sectional area of 12×1.5 mm for parallel deposition, as shown in Figure 1, or is expanded

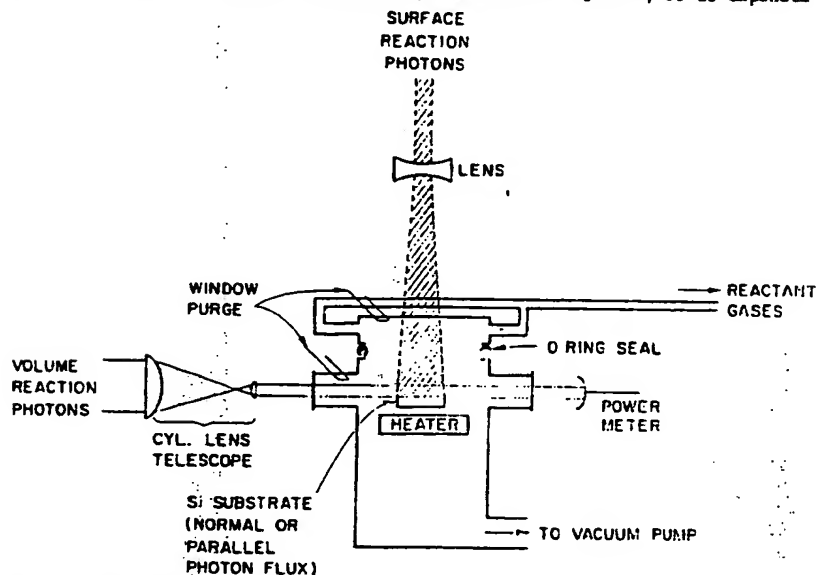


Fig. 1. Experimental Set-up.

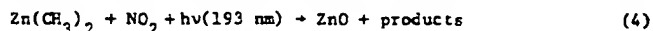
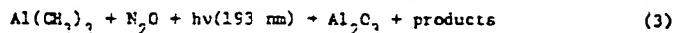
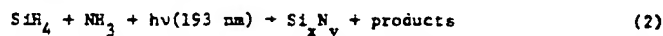
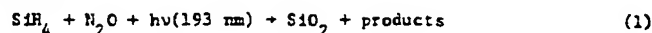
using a negative lens to deposit over large areas during normal irradiation. The insulating films in this work were deposited using a wavelength of 193 nm (ArF^* transition) while the metallic films were deposited using either a 193 nm or the 248 nm (KrF^*) wavelength; repetition rate for each wavelength was 90-100 Hz. UV transmitting windows were purged with a rare gas to prevent deposition on them. Surface reaction photons, whose role is described herein, were provided by a low pressure mercury lamp or by folding back the portion of the beam transmitted through the cell. A substrate heater capable of heating up to 500°C was used during the deposition of dielectric films.

A major advantage of this experimental scheme is the ability to vary laser power, wavelength, and spatial location independently while not affecting the deposition process. This is in direct contrast to plasma-enhanced CVD where process parameters are strongly interrelated and one is limited to pressure regimes where a discharge can be started and maintained. The only pressure constraint on the laser CVD technique is that the gases used for a given deposition must be at optically thin concentrations. Thus beam attenuation across a sample is minimized and thickness uniformity is preserved. For example, deposition of SiO_2 is possible at total pressures up to 8 Torr since the beam intensity will vary by less than 10% across a 3 inch wafer at this pressure.

Additionally, conformal step coverage is possible by this technique due to the photodissociation volume being an "infinite plane source" with respect to topographical features on the sample. This is demonstrated herein.

DEPOSITION OF OXIDE AND NITRIDE FILMS

Films of SiO_2 , Si_3N_4 , Al_2O_3 and ZnO have been deposited via the following reactions, respectively:



The silicon-compound insulator films are compared to the competitive low-temperature deposition schemes of plasma-enhanced and mercury photosensitized CVD, while the metal oxides are discussed on the basis of their properties alone.

Typical deposition conditions for deposition of SiO_2 are shown in Table Ia. A relatively high reactant gas ratio $\text{N}_2\text{O}/\text{SiH}_4$ was used in this work.

TABLE I
Typical Deposition Conditions

| | Laser | RF Plasma ¹ | |
|-----------------------------------|---|------------------------|-----------------|
| | | 380°F | Hg Photoreactor |
| a) SiO_2 | | | |
| Substrate temperature | 150-400°C | 380°F | 100-200°C |
| Cell pressure | 6 Torr | 1.1 Torr | 0.3-1 Torr |
| $\text{N}_2\text{O}/\text{SiH}_4$ | 73 | 33 | 25 |
| Deposition rate | 600 Å/min (He buffer) 800 Å/min (N_2 buffer) | 300 Å/min | 120 Å/min |
| b) Si_3N_4 | | | |
| Substrate temperature | 200-425°C | 380°C | 150-200°C |
| Cell pressure | 2 Torr | 2 Torr | 4 Torr |
| NH_3/SiH_4 | 1 | 7 | 30 |
| | 10 SCCM NH_3 10 SCCM SiH_4 50 SCCM N_2 | | |
| Deposition rate | 700 Å/min | 350 Å/min | 65 Å/min |
| c) Al_2O_3 | | | |
| Substrate temperature | 100-400°C | 150-400°C | |
| Cell pressure | 1 Torr | 10 ⁻² Torr | |
| Deposition rate | 1500 Å/min (up to 1 μ/min) | 350 Å/min | |

¹ Reactor manufactured by ASM, Phoenix, AZ.

² R. S. Nowicki, J. Vac. Sci. Tech. 14(1), 127 (1977).

Deposition rates (which are for films of area ~20 cm²) could be increased using a higher silane density since this reactant is optically transparent at 193 nm. Deposition rates of ~5000 Å/min have been observed over areas of ~3 cm².

The electrical, chemical and physical properties of the laser CVD SiO_2 films have been measured. The films are comparable to plasma and photosensitized CVD films with respect to adhesion, stress, index of refraction stoichiometry,

and hydrogen incorporation as shown in Table II. The laser deposited films are

TABLE II
Comparison of Deposited SiO_2 Films

| | Laser CVD | Plasma | Hg Photox |
|--|----------------------------|----------------|---------------------|
| Breakdown voltage (MV/cm) | 6.5-8 (1000 Å film) | 10 (2000 Å) | 4-8 (1000-10,000 Å) |
| Resistivity ($\Omega\text{-cm}$) at 5 MV/cm | up to 6.7×10^{13} | 10^{16} | 2×10^{12} |
| Stoichiometry | SiO_2 | SiO_2 | $\text{SiO}_{1.9}$ |
| H content (by IR) | | | |
| as Si-H (at.%) | 1-4 | <4 | None |
| as Si-OH (at.%) | <1 | <1 | None |
| Etch Rate in 5:1 BHF | >55 | 22 (7:1 BHF) | 140 |
| Refractive index | 1.48 | 1.49 | 1.46 |
| Stress on Si (10^9 dyne/cm ²) (all compressive) | 1.5 | 3.6 | 2 |

inferior compared to plasma-enhanced CVD films in terms of electrical resistivity (~ 100 times lower), dielectric strength (20% lower breakdown voltage), and etch rate in a buffered oxide etch ($\sim 2 \times$ faster). Laser CVD films exhibited the lowest internal stress and pinhole densities. A 1000 Å SiO_2 film photo-deposited at 400°C had no pinholes in 5 cm², while a 2000 Å plasma CVD film showed $<1/\text{cm}^2$ and a 10,000 Å photox SiO_2 film had 5 to 10 per square centimeter. In the laser CVD approach conformal step coverage is achieved over a wide range of deposition conditions. Figure 2 shows SiO_2 (~ 5000 Å thick) photodeposited over a 4000 Å polysilicon step which was formed on a oxidized silicon wafer. It should be noted the rough surface atop the step is due to the underlying poly-Si while the even morphology of the oxide is retained below the step, over smooth oxidized Si wafer.



Fig. 2. Step Coverage of SiO_2 over 4000 Å Polysilicon Step.

osited films are

Hg Photox

4-E (1000-
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SiO₂ 1.9

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Electrical resistivity (down voltage),
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Silicon nitride has also been deposited using laser-induced CVD using the reaction in equation (2). The conditions for deposition are similar to the two techniques under comparison and to those of the SiO₂ deposition process discussed above (Table Ib). A notable difference is that lower concentrations of ammonia are required since its absorption cross-section is $\sim 10^3$ higher than nitrous oxide at 193 nm [3]. Deposition rate is still much higher than that of plasma or mercury sensitized reactions.

Again the photodeposited films have comparable physical properties (i.e. adhesion, compressive stress, refractive index, step coverage and stoichiometry) as shown in Table III. Pinhole densities are comparable to plasma CVD films

TABLE III
Comparison of Deposited Silicon Nitride Films

| | Laser CVD <SiN | Plasma >SiN | Hg Photride Variable |
|---------------------------------|--------------------|------------------|-------------------------|
| Stoichiometry | | | |
| Impurities (at. %) | | | |
| H by IR-as Si-H | 12 | 12-16 | ----- |
| as N-H | 11-20 | 2-7 | <"typical plasma" |
| O by ESCA | <5 | ----- | ----- |
| Etch rate (Å/sec) in 5:1 BOE | 15 (dep. at 425°C) | 1.7 (7:1 BOE) | 12 |
| No surface photons | 44 (dep. at 380°C) | ----- | ----- |
| 254 nm photons | 27 | ----- | ----- |
| 193 nm photons | 8 | ----- | ----- |

but superior to photride films. The laser CVD films lack in terms of etch rates; they etch approximately ten times faster than plasma deposited nitrides. However, at the bottom of Table III the effect of low level surface irradiation can be seen. As deposited at 380°C, the laser CVD silicon nitride films etched at 44 Å/sec in 5:1 buffered oxide etch, indicative of a porous or low density film. Illumination during deposition with 254 nm photons from a low pressure mercury discharge lamp reduced this etch rate to 27 Å/sec. This etch rate was reduced further to 8 Å/sec by folding back the transmitted portion of the 193 nm dissociating laser beam. The power density of both on the substrate surface was weak so as not to cause surface heating. Clearly, a surface reaction is occurring but has not been modeled at this time.

Using this technique, oxides of aluminum and zinc have been deposited but are not as fully characterized at this time. Conditions for Al₂O₃ growth are tabulated in Table Ic. As compared to films obtained by RF plasma deposition, the laser CVD Al₂O₃ films shown comparable adhesion, stress, stoichiometry, and refractive index. As with the silicon compounds discussed previously, the photodeposited films show higher etch rate ($\times 10$) but have a low pinhole density (none in 5 cm² for an 1100 Å laser CVD film versus 36/cm² for a 2500 Å plasma deposited film). The only other major difference known presently is that the photodeposited films have shown up to 1% carbon contamination, probably due to dissociation of methyls in the aluminum donor gas. The effect of this impurity on the electrical properties is not known at this time.

To obtain ZnO, an oxygen donor, either NO₂ or N₂O was introduced into the cell together with dimethylzinc (DMZ). At 248 nm laser wavelength (KrF) and using N₂O as the oxygen donor, clear films of ZnO were obtained but at a very

slow deposition rate (5000 Å/min) was obtained with NO_2 irradiated at 193 nm. Best results with respect to both the deposition rate and stoichiometry of the films were obtained with NO_2 flow rate of 34 sccm and DMZ pressure of 30 mTorr. The He window purge flow rate was 100 sccm. An automatic throttling valve maintained the total cell pressure at 2 Torr. The substrate temperatures ranged from room temperature to a maximum of 220°C.

The deposited ZnO films appeared clear. Uniform films over 2 cm x 5 cm area were obtained at deposition rates of over 5000 Å/min. The difference in thickness over 5 cm (end to end) was less than 5%. It should be pointed out that the uniformity of the deposited ZnO films is important for fabrication of surface acoustic wave (SAW) devices [4]. At higher deposition rates ($\sim 1 \mu\text{m/min}$) the films had a tendency to peel when exposed to atmospheric pressure and were nonuniform.

The stoichiometry of the deposited film, by ESAC, showed the films to be composed of 49% Zn and 51% oxygen. Carbon was less than 1% and there was no measurable trace of N. By increasing the ratios of DMZ to NO_2 flows, ZnO stoichiometry was easily changed causing the measured sheet resistivities to range from 10^3 to 10^{-1} ohms/square for 0.5 μ thick films. The refractive index of the stoichiometric film, deposited at 200°C was measured on an ellipsometer to be 1.86. The index of refraction increased with the abundance of Zn in the film and with higher deposition temperatures as expected.

When the film adhesion was measured, in all cases the Si substrates cracked ($\sim 10^7$ dynes/cm²) before the ZnO films could be detached. The stress of the photodeposited ZnO film was determined by the x-ray technique. The stress of a 2000 Å thick ZnO film on a Si wafer was 7×10^9 dynes/cm² tensile. The etch rate in 5:1 buffered oxide etch (BOE) of ZnO deposited at 200°C was found to be < 20 Å/min. The pinhole density of 1000 Å thick film of ZnO deposited at 180°C was measured on a Gasconics pinhole monitor to be $< 1 \text{ cm}^2$.

DEPOSITION OF METALS

We have previously reported laser-induced deposition of refractory metals over small areas (10^{-4} cm^2) [5]. As an extension of our earlier work we have investigated large area ($> 5 \text{ cm}^2$) photodeposition of Al, Mo, W and Cr. Uniform films of these metals were deposited on pyrex and quartz substrates as well as silicon wafers at room temperature. We have examined the resistivity, adhesion, stress, and step coverage of these films.

Plasma assisted CVD of refractory metals occurs as low as 350°C (nm/min deposition rates) for refractory halides [6] but plasma parameters such as rf power and frequency, gas flow, electrode spacing, total pressure and substrate heating are all interrelated and difficult to control individually. Photodissociation occurs only along the path of the laser beam, unlike plasma excitation, therefore there is less impurity generation from the walls due to plasma ion bombardment. Moreover, the cracking pattern is less complex in photodissociation and hence we have better control and repeatability of deposition conditions.

Our experimental arrangement is shown in Figure 1. All substrates were precleaned in HF and deionized water prior to deposition. The substrates were held either parallel or normal to the incident laser beam. Either a reservoir containing the carbonyl or a flask of trimethylaluminum (TMA) was connected to the cell. For the carbonyls, both the reservoir and the pyrex connecting tube were heated with a heater tape to about 50°C.

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The substrate was first placed into its holder and the cell pumped down with a roughing pump to a few microns. The laser then irradiated the substrate to preclean the surface with the UV radiation; this improves the adhesion of the deposited films. The vacuum pump was then throttled to reduce the cell throughput and the donor gas introduced into the cell. The deposited films appeared as bright silvery films. When the beam was parallel to the substrate, black particulate films of columnar growth resulted, as shown in Figure 3. For this reason, all the films characterized were obtained at



Fig. 3. Columnar Grain Growth of Chromium Film. Magnification is 20,000X.

normal incidence. Thick (>1 μ) Cr and Mo films deposited at room temperature had a tendency to peel when exposed to air. This could be avoided by heating the substrate to about 150°C during deposition or prior to removal from the cell. All the photodeposited films discussed below were obtained at room temperature.

The purity of the photodeposited films was examined by Auger and ESCA analysis. The major impurity in all the films was oxygen (<7%) probably due to the relatively poor vacuum obtained with a roughing pump. We hope to reduce this impurity by using a improved deposition cell and a better vacuum system. A surprising result was the relatively low concentration of carbon in these films (Table IV). The most carbon-free films and the highest deposition rates were obtained using a laser wavelength of 248 nm; 0.25, 0.17, 0.10 and 0.2 μ /min deposition rates were measured for Mo, W, Al and Cr, respectively, over 2.5 x 2.5 cm area. But even this low contamination by carbon can limit the obtainable film resistivity [7]. These rates will vary with the laser power, the cell pressure, and the size of the area over which the film is deposited. The film over the 2.5 x 2.5 cm area was uniform to $\pm 15\%$. It should be pointed out that the area of deposition can be varied by changing the divergence of the laser beam with a lens; with tight focusing and substrate or beam translation, patterned lines can be deposited.

The adhesion of the photodeposited films was measured. In the case of W, the machine reached its upper limit without detaching the films, while in the case of Al, Mo and Cr, the quartz substrates chipped off before the films were detached. Of the four metals, Cr films were the least adhesive while W were the best, which remained intact even when placed in an ultrasonic cleanser. The most adhesive films were deposited using the 193 nm laser wavelength for photodissociation. The reasons for the change of carbon content and adhesion

with the laser wavelength are not fully understood at this time. Stress measurements of the photodeposited films were made by the substrate bending technique. The metal films were deposited on microscope cover slips and the bowing caused by the films was measured. All the films had tensile stress and none was higher than 7×10^9 dynes/cm².

The electrical resistivities of the deposited metal films were measured with a four-point probe. These resistivities are tabulated along with the bulk values in Table IV. These resistivities are at most about a factor of 20 higher than bulk resistivity values, while the aluminum had, even with its high carbon content, a resistivity approaching the bulk value.

TABLE IV
Summary of the Physical Properties of the Laser Deposited Al, Mo, W and Cr Films

| | Deposition Rate (Å/min) | Resistivity (μΩ-cm) | | Percent Carbon in Film | Adhesion, on quartz (dynes/cm ²) | Tensile Stress (dynes/cm ²) |
|----|-------------------------------|------------------------|------|------------------------------|--|---|
| | | bulk | film | | | |
| Mo | 2500 | 5.2 | 36 | <0.9 | $>5.5 \times 10^8$ | $<3 \times 10^9$ |
| W | 1700 | 5.65 | 135 | <0.7 | $>6.5 \times 10^8$ | $<2 \times 10^9$ |
| Cr | 2000 | 12.9 | 210 | <0.8 | $>5.4 \times 10^8$ | $<7 \times 10^9$ |
| Al | 1000 | 2.66 | 3.0 | <4.0 | $>5.5 \times 10^8$ | $<1 \times 10^9$ |

One important quality of a film deposition technique is the ability of the deposited film to cover vertical-walled steps. Step coverage patterns used to check our deposited refractory films were the same as those used to examine SiO₂ step coverage. The photodeposited refractory metal thicknesses were varied between 0.2 and 0.6μ. After deposition, the metal coated wafers were chilled in liquid nitrogen and then cleaved. The step coverage was examined with a SEM. An ~5000 Å Al film is shown in Figure 4. It can be seen that the film is of

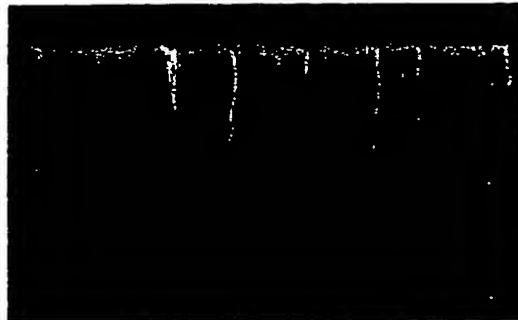


Fig. 4. Step Coverage of 5000 Å Aluminum Film Over 4000 Å Polysilicon Step.

even thickness over the flat, as well as the vertical walls, and clearly demonstrates conformal step coverage. The vertical striations in the films are due to wafer cleaving. It is interesting to note that SEM examination of all

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W and Cr Films

Tensile
Stress
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our films showed absence of microstructures similar to those seen in laser photodeposited Cd and Zn films [8].

SUMMARY

We have described a technique to deposit oxide, nitride, and metal films via ultraviolet photolysis of gas-phase donor molecules. All films are deposited at fast rates (up to 5000 Å/min) and demonstrate conformal step coverage over vertical steps. The insulating films exhibit low pinhole densities but are inferior in terms of etch rate and electrical resistivity. Surface photon impingement during film deposition is shown to drastically reduce silicon nitride etch rate. Future work will focus in this area. Metallic films have been deposited and exhibit good physical properties. The refractory metals show high resistivities which may be limited by carbon incorporation. The effect of annealing these films has not been studied yet.

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